Table 2. *Selected geometric parameters (Å, °) for (2)*

S—C5	1.634 (1)	N4—C3	1.384 (1)
O1—N2	1.421 (1)	N4—C5	1.360 (2)
O1—C5	1.350 (2)	C3—C6	1.473 (2)
N2—C3	1.298 (2)		
N2—O1—C5	109.7 (1)	N2—C3—N4	112.0 (1)
O1—N2—C3	104.60 (9)	O1—C5—N4	106.86 (9)
C3—N4—C5	106.8 (1)		
C12—N4—C3—C6	1.1 (2)	N4—C3—C6—N7	3.3 (2)
C12—N4—C5—S	−0.1 (2)	N4—C12—C13—C14	170.0 (1)
C3—N4—C12—C13	−86.6 (2)		

The H atoms of (1) were placed in calculated positions with C—H 0.93 Å and isotropic displacement parameters at $1.2U_{eq}$ of the bonded C atom. H atoms of the methyl group at C19 were calculated with C—H 0.96 Å in idealized positions of maximum total electron density. A torsional parameter specifying the conformation of the methyl group was refined. All other H atoms were allowed to ride on the attached C atom. H atoms of (2) were refined with isotropic displacement parameters. C—H distances are in the range 0.87 (2)–1.01 (2) Å, while U_{iso} values are in the range 0.024 (5)–0.042 (7) $Å^2$.

For both compounds, data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *EXPRESS*. Data reduction: *XCAD*4 (Harms & Wocadlo, 1996) for (1); *PROCESS* in *MolEN* (Fair, 1990) for (2). Program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990) for (1); *SIR* (direct methods) (Burla *et al.*, 1989) for (2). Program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993) for (1); *LSFM* in *MolEN* for (2). For both compounds, molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXTL* for (1); *CIF IN* in *MolEN* for (2).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DA1041). Services for accessing these data are described at the back of the journal.

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N-Methyl-2′-nitrocinnamanilide

E. Subramanian,[a] S. Renganayaki,[a] S. Shanmuga Sundara Raj[b] and Hoong-Kun Fun[b]

[a]*Department of Crystallography and Biophysics, University of Madras, Guindy Campus, Chennai 600 025, India, and* [b]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: crystal@giasmd01.vsnl.net.in*

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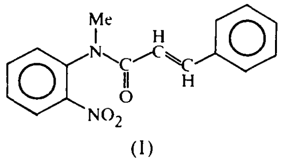
Abstract

An inversion centre relates two molecules of the title compound, $C_{16}H_{14}N_2O_3$, which form a dimer through C—H···O hydrogen bonds. The inversion centre lies almost in the plane of the dimer. This study provides a good example of C—H···O interactions leading to dimeric coupling of molecules.

Comment

The title compound, (I), is an intermediate in the synthesis of substituted indole steroids. An interesting aspect of the structure is the occurrence of an inversion-related dimer linked by C—H···O hydrogen bonds.



(I)

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Fig. 1. shows the conformation and the displacement ellipsoids for a molecule of (I). The dimer acts as the basic building block whose packing leads to the observed crystal structure. A stereoscopic view of the dimeric coupling through C—H···O bonds is shown in Fig. 2. Similar to the case in the crystal structures of carboxylic acids forming cyclic dimers, a third C—H···O hydrogen bond (*viz.* C17—H17···O10) relates the dimer to other molecules in the lattice. This study demonstrates clearly that C—H···O contacts represent bonding interactions. Atoms of the dimer involved in the loop formed by the hydrogen bonds all lie in the same plane.

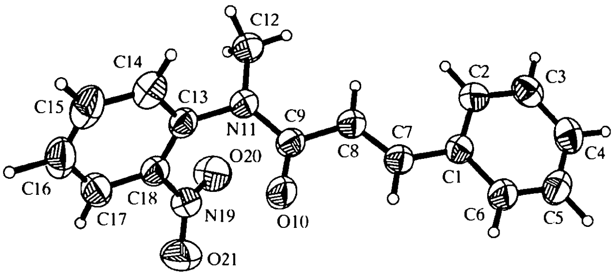

Fig. 1. The structure of a molecule of (I) showing 50% probability displacement ellipsoids and the atom-numbering scheme.

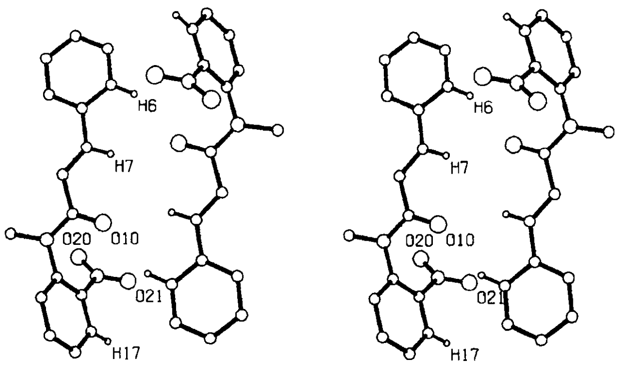

Fig. 2. A stereoscopic view of a dimer in (I), which is linked by C—H···O hydrogen bonds (not drawn for clarity).

Experimental

The synthesis of (I) was carried out by dissolving pyridine and *o*-nitroaniline in dichloromethane and adding cinnamoyl chloride in drops to the reaction mixture. Once the reaction was complete, dichloromethane was distilled off and the reaction mixture poured into water. The product was purified by repeated crystallization from a mixture of hexane and dichloromethane.

Crystal data

$C_{16}H_{14}N_2O_3$
$M_r = 282.29$
Monoclinic
$P2_1/n$
$a = 10.387\,(1)$ Å
$b = 10.248\,(1)$ Å
$c = 13.857\,(3)$ Å
$\beta = 107.54\,(2)°$
$V = 1406.5\,(1)$ Å^3
$Z = 4$
$D_x = 1.333$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 5465 reflections
$\theta = 2.86–33.16°$
$\mu = 0.094$ mm^{-1}
$T = 293\,(2)$ K
Block
0.62 × 0.48 × 0.40 mm
Yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
9043 measured reflections
3213 independent reflections

2493 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$
$\theta_{\rm max} = 27.5°$
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.124$
$S = 1.029$
3213 reflections
246 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.2854P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.299$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.150$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

C1—C7	1.467 (2)	N11—C13	1.435 (2)
C7—C8	1.329 (2)	N11—C12	1.460 (2)
C8—C9	1.485 (2)	C18—N19	1.482 (2)
C9—O10	1.227 (2)	N19—O21	1.211 (2)
C9—N11	1.359 (2)	N19—O20	1.215 (2)
C5—C6—C1	120.84 (15)	C9—N11—C13	117.30 (11)
C8—C7—C1	126.37 (13)	C9—N11—C12	126.93 (13)
C7—C8—C9	120.12 (13)	C13—N11—C12	115.71 (11)
O10—C9—N11	120.32 (13)	O21—N19—O20	125.0 (2)
O10—C9—C8	121.37 (12)	O21—N19—C18	117.02 (15)
N11—C9—C8	118.31 (12)	O20—N19—C18	118.00 (13)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O10$^{\rm i}$	0.96 (2)	2.49 (2)	3.388 (2)	155 (2)
C7—H7···O10$^{\rm i}$	0.95 (2)	2.56 (2)	3.447 (2)	155 (1)
C17—H17···O10$^{\rm ii}$	0.95 (2)	2.53 (2)	3.386 (2)	150 (2)

Symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $-\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$.

The data collection covered over a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 10 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections; it was found to be negligible. All of the H atoms were located from the difference Fourier map and refined isotropically.

Data collection: *SMART* (Siemens, 1996*a*). Cell refinement: *SAINT* (Siemens, 1996*b*). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: NA1384). Services for accessing these data are described at the back of the journal.

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Pipemidic acid hydrochloride†

Hoong-Kun Fun,[a] Kandasamy Chinnakali,[a]‡ Ibrahim Abdul Razak,[a] Shu-zhong Zhan,[b] Chuan-jiang Hu[b] and Qingjin Meng[b]

[a]*X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*The State Key Laboratory of Coordination Chemistry, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@usm.my*

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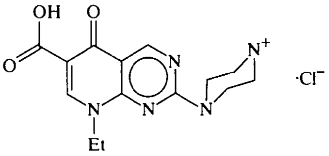
Abstract

The title compound exists as a $C_{14}H_{18}N_5O_3^+$ cation and a Cl^- anion. The apical N atom of the piperazine ring shows quaternary character due to proton transfer from HCl. The fused pyridine and pyrimidine rings are nearly coplanar and the piperazine ring adopts a chair conformation. The carboxyl and carbonyl O atoms are involved in an O—H···O intramolecular hydrogen bond and the Cl^- anion is involved in an N—H···Cl intermolecular hydrogen bond with the quaternary N atom.

Comment

Metal ions play a vital role in intricate biological processes. The interaction of these ions with drugs administered for therapeutic reasons is a subject of considerable interest. It is known that some drugs act *via* chelation (Albert, 1979) or by way of inhibiting the formation of metallo enzymes (Hughes, 1981). For most of the drugs that could act as potential ligands little is known about the metal-binding influences on their activities. In order to understand drug–metal ion interactions further, we have been studying pipemidic acid and its analogues. These antibiotics are used in the clinical treatment of urinary-tract infections caused by Gram-negative bacteria (Shen & Pernat, 1980). The X-ray structure determination of the title compound, (I), was carried out in order to elucidate the molecular conformation.



(I)

The asymmetric unit consists of a $C_{14}H_{18}N_5O_3^+$ cation and a Cl^- anion. The piperazine N atom, N17, shows quaternary character due to proton transfer from HCl. This is in contrast with the 'zwitterion' structure formed in pipemidic acid trihydrate (Fonseca *et al.*, 1986) where

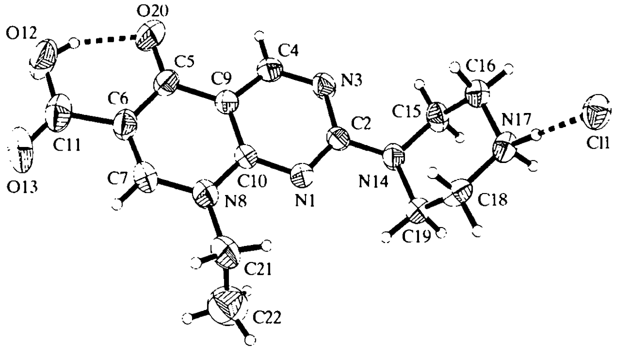


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

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† Alternative name: 4-(6-carboxy-8-ethyl-5,8-dihydropyrido[2,3-*d*]pyrimidin-2-yl)piperazin-1-ium chloride.

‡ On leave from: Department of Physics, Anna University, Chennai 600 025, India.

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